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Lustig

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(54) **METHOD FOR REMOVING CONTAMINANTS FROM BORON POWDER**

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(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(57) **ABSTRACT**

Methods for removing an organic contaminant from contaminated boron powder are provided. One method includes providing a contaminated boron powder, the boron powder being comingled with an organic contaminant. The method also includes placing the contaminated boron powder and the organic contaminant comingled therewith onto an inert container. The method includes placing the inert container, the contaminated boron powder, and the organic contaminant comingled therewith, into an enclosed space. A heat source is provided in the enclosed space. The method also includes heating the contaminated boron powder and the organic contaminant comingled therewith to an elevated temperature. The method includes altering the organic contaminant so as to reduce the amount of the organic contaminant comingled with the boron powder. Another method includes reducing the amount of the organic contaminant comingled with the boron powder to not more than about 0.1 weight percent of soluble residue.

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(51) **Int. Cl.**
C01B 35/02 (2006.01)

(52) **U.S. Cl.** **423/298**

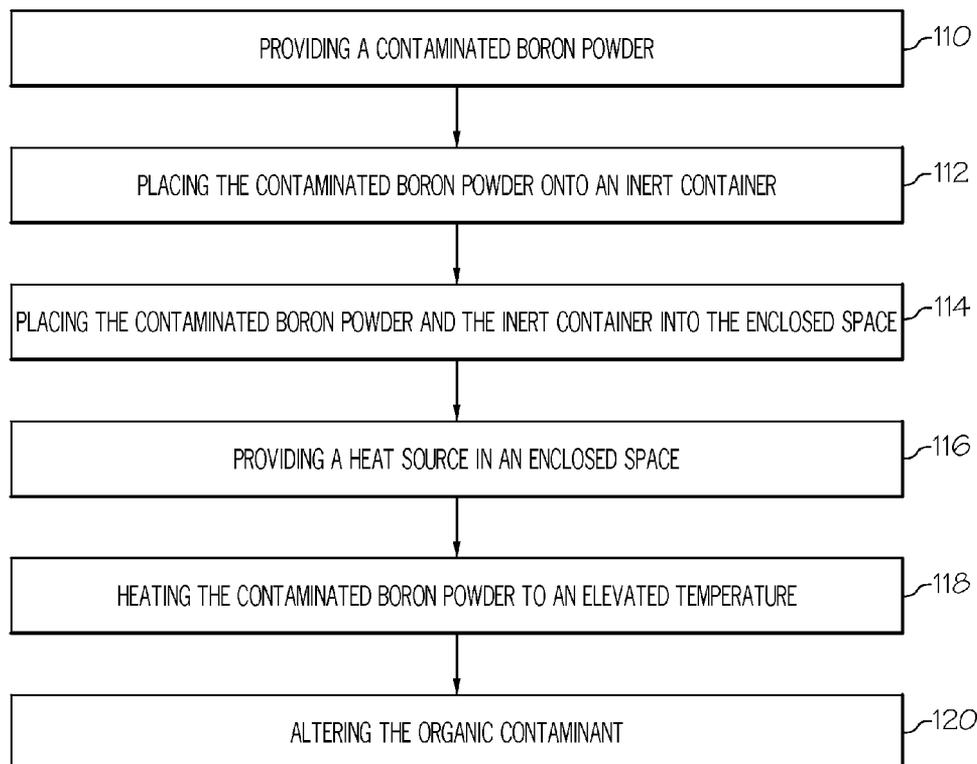
(58) **Field of Classification Search** 423/298
See application file for complete search history.

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12 Claims, 3 Drawing Sheets



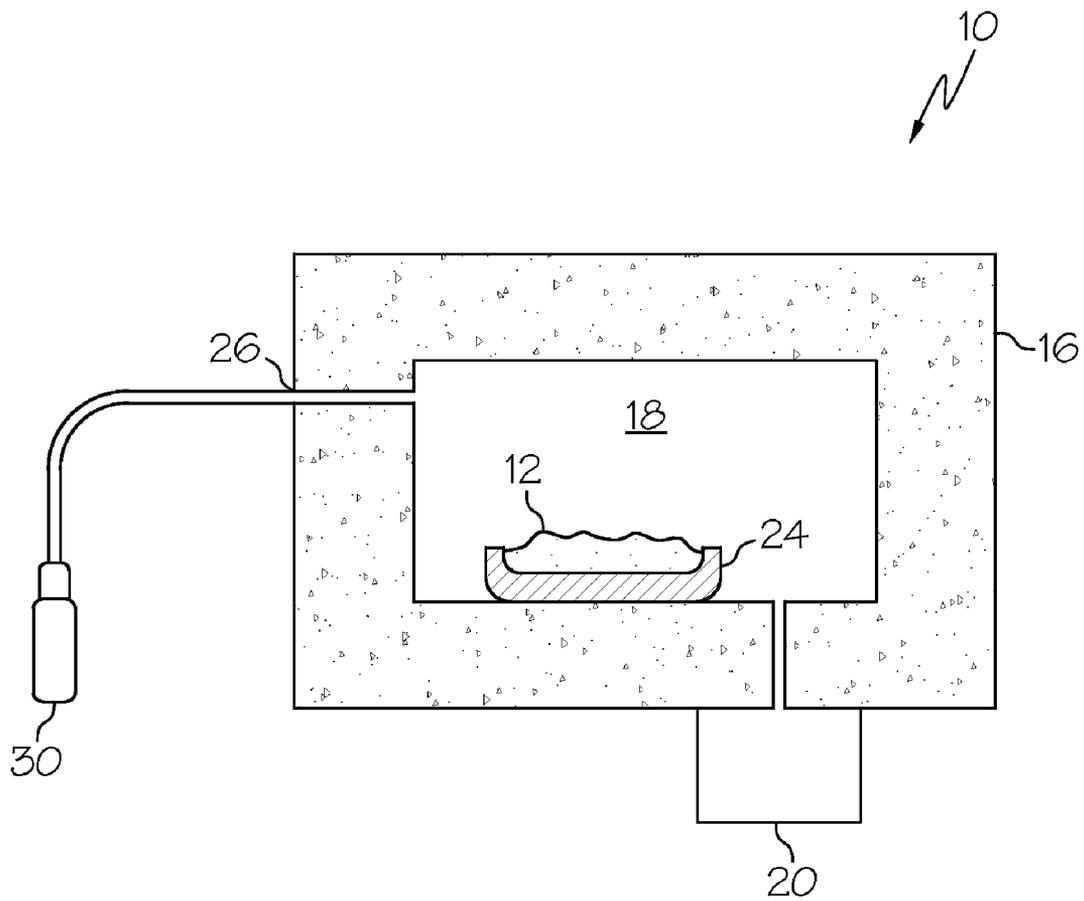


FIG. 1

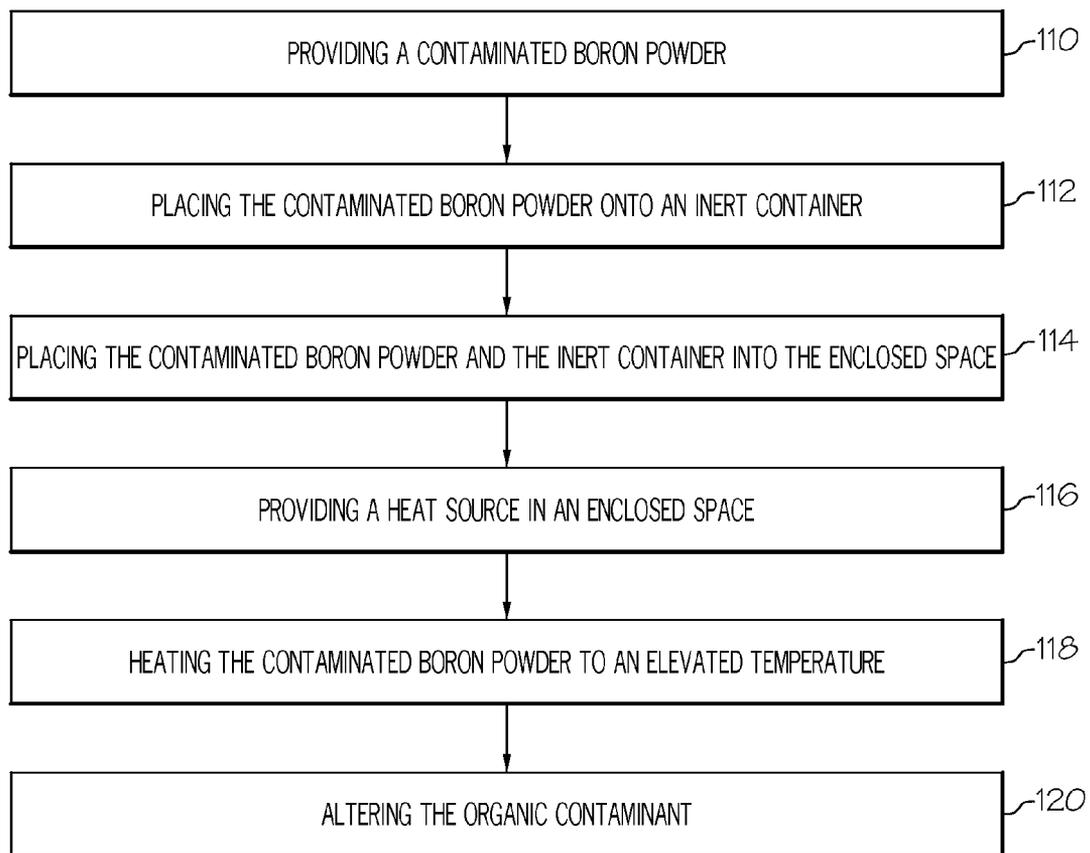


FIG. 2

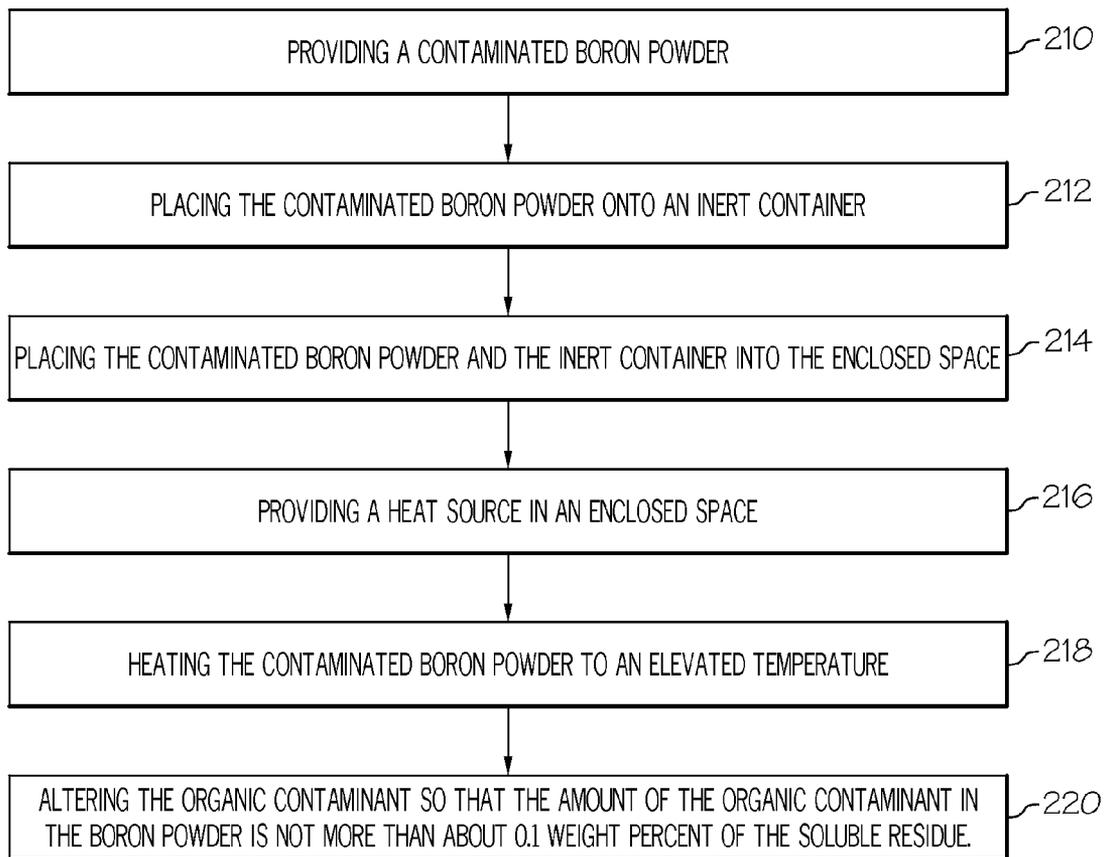


FIG. 3

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METHOD FOR REMOVING CONTAMINANTS FROM BORON POWDER

BACKGROUND OF THE INVENTION

1. Field of the Invention

The subject matter disclosed herein relates to removing contaminants from boron powder.

2. Discussion of the Prior Art

Boron powder is used as a primary component of boron coatings in numerous applications. Such applications include, but are not limited to boron coatings used for neutron detection, abrasion protection for die-casting dies, improved wear resistance for biomedical implants, etc. Some of these applications are adversely affected by contaminants within the boron powder, as the contaminants can be detrimental to boron coating applications.

A contaminated boron powder can include organic contaminants from various sources. For example, jet milled boron powder has been found to be susceptible to contamination from the air supply used in the milling process. Specifically, boron powder contaminants may include lubrication oil from an air compressor when compressed air is used to operate a jet mill. This contamination can result in coating defects such as non-uniform coatings and gas contamination resulting in degraded coating properties. Other example contaminants are polymeric liner material from the jet mill, adhesive materials used to attach the polymeric liner material to a jet mill interior wall, and metal particles from the jet mill interior wall.

Boron powder is a relatively expensive material which, in turn, makes both contaminated boron powder and coated goods costly missteps in the manufacturing process. Some previous methods of recovering contaminated boron powder include rinsing the powder with hexane, methylene chloride, and ethylene glycol, each in combination with filters and/or centrifuges. Therefore, there is a need for an improved apparatus and method of removing contaminants from the surfaces of boron powder particles.

BRIEF DESCRIPTION OF THE INVENTION

The following presents a simplified summary of the invention in order to provide a basic understanding of some example aspects of the invention. This summary is not an extensive overview of the invention. Moreover, this summary is not intended to identify critical elements of the invention nor delineate the scope of the invention. The sole purpose of the summary is to present some concepts of the invention in simplified form as a prelude to the more detailed description that is presented later.

In accordance with one aspect, the present invention provides a method of removing a contaminant from contaminated boron powder. The method includes providing a contaminated boron powder, the boron powder being comingled with an organic contaminant. The method also includes placing the contaminated boron powder and the contaminant comingled therewith onto an inert container. The method includes placing the inert container, the contaminated boron powder, and the contaminant comingled therewith, into an enclosed space. A heat source is provided in the enclosed space. The method also includes heating the contaminated boron powder and the contaminant comingled therewith to an elevated temperature. The method includes altering the contaminant so as to reduce the amount of the organic contaminant comingled with the boron powder.

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In accordance with another aspect, the present invention provides a method of removing a contaminant from contaminated boron powder. The method includes providing a contaminated boron powder, the boron powder being comingled with an organic contaminant. The method also includes placing the contaminated boron powder and the contaminant comingled therewith onto an inert container. The method includes placing the inert container, the contaminated boron powder, and the contaminant comingled therewith, into an enclosed space. A heat source is provided in the enclosed space. The method also includes heating the contaminated boron powder and the contaminant comingled therewith to an elevated temperature. The method includes altering the contaminant so that the amount of the organic contaminant in the boron powder after a processing cycle is not more than about 0.1 weight percent of soluble residue.

BRIEF DESCRIPTION OF THE DRAWINGS

The foregoing and other aspects of the present invention will become apparent to those skilled in the art to which the present invention relates upon reading the following description with reference to the accompanying drawings, in which:

FIG. 1 is a schematized cross section view of an example furnace of an example processing system in accordance with an aspect of the present invention;

FIG. 2 is a top level flow diagram of an example method of removing organic contaminants from boron powder in accordance with an aspect of the present invention; and

FIG. 3 is a top level flow diagram of an example method of removing organic contaminants from boron powder in accordance with an aspect of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

Example embodiments that incorporate one or more aspects of the present invention are described and illustrated in the drawings. These illustrated examples are not intended to be a limitation on the present invention. For example, one or more aspects of the present invention can be utilized in other embodiments and even other types of devices. Moreover, certain terminology is used herein for convenience only and is not to be taken as a limitation on the present invention. Still further, in the drawings, the same reference numerals are employed for designating the same elements.

An example processing system **10** for removing contaminants from boron powder **12** is generally shown within FIG. 1. In one specific example, the processing system **10** is for removing organic contaminants from boron powder **12**. It is to be appreciated that the term organic is a broad and expansive classification. In one part, the classification includes materials that contain a carbon component. It is also to be appreciated that FIG. 1 merely shows one example of possible structures/configurations/etc. and that other examples are contemplated within the scope of the present invention.

The processing system **10** for removing organic contaminants from contaminated boron powder **12** includes a furnace **16**, which is one example of an enclosed space. Other examples of an enclosed space include, but are not limited to batch ovens, continuous ovens, cabinet ovens, tower ovens, sintering furnaces, etc. It is to be appreciated that an interior volume **18** of the furnace **16** can be secured so that little or no ambient atmosphere can enter into the furnace during operation of the furnace. Selection of the type of furnace **16** and construction thereof is dependent upon several variables including, but not limited to, furnace heating characteristics, furnace cycle times, boron powder throughput requirements,

etc. The furnace **16** also includes a heat source **20** to provide the elevated temperature within the furnace **16**. The heat source **20** can be any of the typical furnace **16** or oven heat sources as are known in the art such as gas, electric heating element, infrared, microwave, etc. The heat source **20** is schematically shown and is only schematically shown in position. The structure and position can be suitably selected to heat the interior volume **18**. In any of the examples, the furnace **16** can include an exhaust port that can be used to purge vaporized contaminants from the interior volume **18**.

The interior volume **18** of the furnace **16** provides space for the contaminated boron powder **12**. A furnace heating cycle can begin after the boron powder **12** has been placed into the furnace **16**. The furnace heating cycle subjects the boron powder **12** to an elevated temperature within the furnace **16**. Temperature profiles for the furnace heating cycle may ramp up to a particular temperature, hold constant for a time and then ramp down. However, it is contemplated that the temperature profile may be of various multiple temperatures in order to optimize the heat application to the boron powder **12** and contaminant removal process. In one example, the boron powder **12** is subjected to an elevated temperature of about 500° C. This temperature promotes the vaporization of some organic contaminants, and this temperature can also promote thermal decomposition of some organic contaminants.

The processing system **10** further includes a boat **24**, which is one example of an inert container for holding the boron powder **12** within the furnace **16**. The boat **24** can be made of material that is resistant to the effects of high temperature, numerous heating and cooling cycles, and is not likely to impart contaminants to the boron powder **12** that it contains. Quartz is a common choice as a boat **24** material, as it can have smooth surfaces which promote easy removal of boron powder **12**, it is typically easy to clean, and it has surface characteristics that can make any boron powder **12** remaining in the boat **24** after its intended removal readily visible to the casual observer. Several ceramic compounds are also common choices as a boat **24** material. The boat **24** can be shaped like a rectangular or square bowl, with a horizontal bottom and four vertical sides, although the boat may be constructed of various materials and have varied dimensions and shapes. Boats **24** can be used in batch furnaces or can be used in continuous furnaces, riding a conveyor as they pass through various heating zones.

The processing system **10** can also include at least one port **26** for introducing at least one cover gas **30** (schematically represented by a bottle-type source example) into the furnace **16**. The cover gas **30** can provide a special atmosphere within the furnace **16**, consisting of one gas or a combination of gases. In one example, hydrogen is used as a cover gas **30**. Hydrogen provides a reducing atmosphere within the furnace **16**. One quality of the hydrogen reducing atmosphere is the promotion of the break-down of longer carbon chains into smaller, more easily volatilized organic compounds. This quality is particularly useful in eliminating some of the organic contaminants such as lubrication oil from a typical industrial compressed air supply. This lubrication oil is formulated specifically to be resistant to thermal decomposition. The break-down of the carbon chains that make up the lubrication oil can lead to the vaporization of resultant organic contaminants that are more susceptible to thermal decomposition. Additionally, the hydrogen cover gas **30** minimizes the oxidation of the boron powder **12**. The combination of the hydrogen reducing atmosphere and a furnace **16** temperature of about 500° C. reduces the likelihood of oxidation of the boron powder **12**. Lower oxidation rates tend to eliminate boron coating defects in downstream manufacturing pro-

cesses. Another benefit to the inclusion of a cover gas is that reactive or inert cover gases provide convection. Convection action within the interior volume **18** helps to speed the transfer of heat into the boron powder **12** and also helps to purge any vaporized compounds from the surface of the boron powder **12**.

The processing system **10** can also be used with a cooling cycle after the thermal decomposition of the contaminants. In order to decrease oxidation of the boron powder **12**, the boron powder **12** can be cooled prior to removal from the interior volume **18** and the protective special atmosphere within the interior volume **18**. One example of a cooling cycle includes reduction of the boron powder **12** temperature to less than about 100° C. prior to removing the boron powder **12** from the interior volume **18**.

Removal of the organic contaminants in the boron powder **12** via the break-down of longer carbon chains into smaller, more easily volatilized organic compounds and the vaporization of organic contaminants enables production of a boron powder **12** with not more than about 0.1 weight percent of soluble residue. This level of impurity can be considered to be an acceptable level of soluble residue that does not affect a hydrophilic nature of the boron powder **12**. One solvent that can be used to determine the amount of soluble residue within the boron powder **12** is methylene chloride via methods that are known in the art.

The method of removing organic contaminants from boron powder **12** using a furnace **16** to break-down longer carbon chains and vaporize the organic contaminants and the associated process system is one solution to remove organic contaminants from a boron powder **12**. Additionally, the use of a furnace **16** to remove the organic contaminants is a relatively simple alternative to other chemical wash methods of removing organic contaminants from boron powder **12**. Furthermore, the use of hydrogen as a cover gas **30** in a reducing atmosphere reduces the likelihood of oxidation of the boron powder **12**.

An example method of removing organic contaminants from boron powder **12** to meet purity requirements for downstream manufacturing applications is generally described in FIG. 2. The method can be performed in connection with the example furnace **16** shown in FIG. 1. The method includes the step **110** of providing a contaminated boron powder **12**, the boron powder being comingled with an organic contaminant. The organic contaminants can be introduced to the boron powder **12** during a jet milling operation from sources such as air compressor oils, adhesive materials, and particles of a polymeric liner material used on the interior of a jet mill.

The method includes the step **112** of placing the contaminated boron powder **12** onto a boat **24**, which is one example of an inert container used in processing furnaces **16**. The boat **24** can be made of material that is resistant to the effects of high temperature, numerous heating and cooling cycles, and is not likely to impart contaminants to the boron powder **12** that it contains. Quartz and some ceramic compounds are common choices for boat **24** construction material.

The method includes the step **114** of placing the contaminated boron powder **12** and the inert container into the enclosed space. The method includes the step **116** of providing a heat source **20** for an enclosed space. The heat source **20** can be any one or a combination of typical heat sources such as gas, electric heating element, infrared, microwave, etc. Examples of an enclosed space include, but are not limited to batch ovens, continuous ovens, cabinet ovens, tower ovens, sintering furnaces, etc.

The method also includes step **118** of heating the contaminated boron powder **12** to an elevated temperature. The heat

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source **20** is activated and increases the temperature within the furnace **16**. In one example, the heat source **20** subjects the boron powder **12** within the enclosed space to an elevated temperature of about 500° C.

The method includes the step **120** of altering the organic contaminant so as to reduce the amount of organic contaminant comingled with the boron powder **12**. In one example, the alteration of the organic contaminant is breaking down long carbon chains forming the organic contaminant. A hydrogen cover gas **30** within the furnace **16** provides a reducing special atmosphere that promotes this break-down of long carbon chains. The alteration of the organic contaminant may also be vaporizing the organic contaminant. Often, these two modes of alteration of the organic contaminant work in cooperation to eliminate organic contaminants. Lubrication oil can be an organic contaminant within the boron powder **12**, and it is formulated specifically to be resistant to thermal decomposition. The break-down of the carbon chains that make up the lubrication oil by the reducing atmosphere in the furnace **16** can lead to the vaporization of resultant broken-down organic contaminants that are more susceptible to thermal decomposition.

Another example method of removing organic contaminants from boron powder **12** to meet purity requirements for downstream manufacturing applications is generally described in FIG. 3. The method can be performed in connection with the example furnace **16** shown in FIG. 1. The method includes the step **210** of providing a contaminated boron powder **12**, the boron powder being comingled with an organic contaminant. The organic contaminants can be introduced to the boron powder **12** during a jet milling operation from sources such as air compressor oils, adhesive materials, and particles of a polymeric liner material used on the interior of a jet mill.

The method includes the step **212** of placing the contaminated boron powder **12** onto a boat **24**, which is one example of an inert container used in processing furnaces **16**. The boat **24** can be made of material that is resistant to the effects of high temperature, numerous heating and cooling cycles, and is not likely to impart contaminants to the boron powder **12** that it contains. Quartz and some ceramic compounds are common choices for boat **24** construction material.

The method includes the step **214** of placing the contaminated boron powder **12** and the inert container into the enclosed space. The method includes the step **116** of providing a heat source **20** for an enclosed space. The heat source **20** can be any one or a combination of typical heat sources such as gas, electric heating element, infrared, microwave, etc. Examples of an enclosed space include, but are not limited to batch ovens, continuous ovens, cabinet ovens, tower ovens, sintering furnaces, etc.

The method also includes step **218** of heating the contaminated boron powder **12** to an elevated temperature. The heat source **20** is activated and increases the temperature within the furnace **16**. In one example, the heat source **20** subjects the boron powder **12** within the enclosed space to an elevated temperature of about 500° C.

The method includes the step **220** of altering the organic contaminant so as to reduce the amount of organic contaminant comingled with the boron powder **12** so that the amount of the organic contaminant in the boron powder is not more than about 0.1 weight percent of soluble residue. In one example, the alteration of the organic contaminant is breaking down long carbon chains forming the organic contaminant. A hydrogen cover gas **30** within the furnace **16** provides a reducing special atmosphere that promotes this break-down of long carbon chains. The alteration of the organic contaminant may

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also be vaporizing the organic contaminant. Often, these two modes of alteration of the organic contaminant work in cooperation to eliminate organic contaminants. Lubrication oil can be an organic contaminant within the boron powder **12**, and it is formulated specifically to be resistant to thermal decomposition. The break-down of the carbon chains that make up the lubrication oil by the reducing atmosphere in the furnace **16** can lead to the vaporization of resultant broken-down organic contaminants that are more susceptible to thermal decomposition.

The method can further include the step of cooling the boron powder **12** to less than about 100° C. prior to removal of the boron powder **12** from the enclosed space. In order to decrease the potential oxidation of the boron powder **12**, the boron powder **12** is kept within the enclosed space for a cooling cycle within the special atmosphere. In one example, the special atmosphere is hydrogen gas which decreases the potential oxidation of the boron powder **12**. The boron powder **12** is then cooled to less than about 100° C. before it is removed from the enclosed space. Various cooling profiles are contemplated for the cooling cycle.

In the described examples, the method and apparatus provide a means for cleaning boron powder **12** prior to making a boron powder coating solution, removing any oil films from the surface of the boron powder particles. The removal of organic contaminants in boron powder **12** via the break-down of longer carbon chains into smaller, more easily volatilized organic compounds and the vaporization of organic contaminants enables production of a boron powder **12** with not more than about 0.1 weight percent of soluble residue. This level of impurity can be considered to be an acceptable level of soluble residue that does not affect a hydrophilic nature of the boron powder **12**. Additionally, the resulting boron powder **12** containing fewer or no organic contaminants reduces or eliminates downstream boron powder coating defects and improves the repeatability in the coating process.

This written description uses examples to disclose the invention, including the best mode, and also to enable any person skilled in the art to practice the invention, including making and using any devices or systems and performing any incorporated methods. The patentable scope of the invention is defined by the claims, and may include other examples that occur to those skilled in the art. Such other examples are intended to be within the scope of the claims if they have structural elements that do not differ from the literal language of the claims, or if they include equivalent structural elements with insubstantial differences from the literal language of the claims.

What is claimed is:

1. A method of removing a contaminant from contaminated boron powder, the method including:
 - providing a contaminated boron powder, the boron powder being comingled with an organic contaminant;
 - placing the contaminated boron powder and the contaminant comingled therewith onto an inert container;
 - placing the inert container, the contaminated boron powder, and the contaminant comingled therewith, into an enclosed space;
 - providing a heat source for the enclosed space;
 - heating the contaminated boron powder and the contaminant comingled therewith to an elevated temperature, wherein the elevated temperature is about 500° C.; and
 - altering the contaminant so as to reduce an amount of the organic contaminant.
2. The method according to claim 1, wherein the step of altering the contaminant is breaking down a carbon chain forming the organic contaminant.

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3. The method according to claim 1, wherein the step of altering the contaminant is vaporizing the organic contaminant.

4. The method according to claim 1, wherein the method further includes providing a special selected gas atmosphere within the enclosed space. 5

5. The method according to claim 1, further including the step of cooling the boron powder to less than about 100° C. prior to removal of the boron powder from the enclosed space. 10

6. A method of removing a contaminant from contaminated boron powder, the method including: 10

providing a contaminated boron powder, the boron powder being comingled with an organic contaminant;

placing the contaminated boron powder and the contaminant comingled therewith onto an inert container; 15

placing the inert container, the contaminated boron powder, and the contaminant comingled therewith, into an enclosed space;

providing a heat source for the enclosed space; 20

heating the contaminated boron powder and the contaminant comingled therewith to an elevated temperature; and

altering the contaminant so as to reduce an amount of the organic contaminant; 25

wherein the method further includes providing a selected gas atmosphere within the enclosed space and the special selected gas atmosphere is hydrogen.

7. A method of removing a contaminant from contaminated boron powder, the method including:

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providing a contaminated boron powder, the boron powder being comingled with an organic contaminant;

placing the contaminated boron powder and the contaminant comingled therewith onto an inert container;

placing the inert container, the contaminated boron powder, and the contaminant comingled therewith, into an enclosed space;

providing a heat source for the enclosed space; heating the contaminated boron powder and the contaminant comingled therewith to an elevated temperature, wherein the elevated temperature is about 500° C.; and

altering the contaminant so that the amount of the organic contaminant in the boron powder after a processing cycle is not more than about 0.1 weight percent of soluble residue.

8. The method according to claim 7, wherein the step of altering the contaminant is breaking down a carbon chain forming the organic contaminant.

9. The method according to claim 7, wherein the step of altering the contaminant is vaporizing the organic contaminant.

10. The method according to claim 7, wherein the method further includes providing a special selected gas atmosphere within the enclosed space.

11. The method according to claim 10, wherein the special selected gas atmosphere is hydrogen.

12. The method according to claim 7, further including the step of cooling the boron powder to less than about 100° C. prior to removal of the boron powder from the enclosed space.

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UNITED STATES PATENT AND TRADEMARK OFFICE
CERTIFICATE OF CORRECTION

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DATED : April 2, 2013
INVENTOR(S) : James Michael Lustig

Page 1 of 1

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

In the Claims

Claim 4, Column 7, line 5, delete "qas" and insert --gas--;

Claim 6, Column 7, line 27, delete "qas" and insert --gas--;

Claim 6, Column 7, line 28, delete "qas" and insert --gas--.

Signed and Sealed this
Twenty-third Day of July, 2013



Teresa Stanek Rea
Acting Director of the United States Patent and Trademark Office